

Originator: Kenyon C. Carlson, Manager
ADEQ QA Unit

Contact For
Information: Kenyon C. Carlson, Manager
ADEQ QA Unit

METHOD 505

I. SCOPE AND APPLICATION:

This method is used for the analysis of organohalide pesticides and commercial polychlorinated biphenyl (PCB) products in water using microextraction and gas chromatography. This method is applicable to the following analytes:

| <u>Analyte</u> | <u>Chemical Abstract Services Registry Numbers (CASRN)</u> |
|---------------------------|---|
| Alachlor | 5972-60-8 |
| Aldrin | 309-00-2 |
| Atrazine | 1912-24-9 |
| Chlordane | 57-74-9 |
| alpha-Chlorodane | 5103-71-9 |
| gamma-Chlorodane | 5103-74-2 |
| Dieldrin | 60-57-1 |
| Endrin | 72-20-8 |
| Heptachlor | 76-44-8 |
| Heptachlor Epoxide | 1024-57-3 |
| Hexachlorobenzene | 118-74-1 |
| Hexachlorocyclopentadiene | 77-74-4 |
| Lindane | 58-89-9 |
| Methoxychlor | 72-43-5 |
| cis-Nonachlor | 39765-80-5 |
| trans-Nonachlor | 39765-80-5 |
| Simazine | 122-34-9 |
| Toxaphene | 8001-35-2 |
| Arochlor 1016 | 12674-11-2 |
| Arochlor 1221 | 11104-28-2 |
| Arochlor 1232 | 11141-16-5 |
| Arochlor 1242 | 53469-21-9 |
| Arochlor 1248 | 12672-29-6 |
| Arochlor 1254 | 11097-69-1 |
| Arochlor 1260 | 11096-82-5 |

II. REAGENTS:

- Sodium Thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution

III. MATERIALS:

- 40 ml amber vial with plastic screw on caps and Teflon septa.
- Latex gloves
- Paper towels and Kim Wipes
- Pool and Spa 3-Way Test Strips (Chem Lab Products, Inc.)
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Pliers
- Protective eyewear

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.
2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.
3. Adjust the water flow to about 500 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.
4. Remove the cap from the 40 ml container. Do not rinse the container as it has already been acid rinsed and may already contain sodium thiosulfate as a preservative.
5. To fill, tip the vial at about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the vial to the mouth to form a meniscus.
6. Remove the bottle from the flow and recap. Invert the container five times.
7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

IV. PROCEDURE (continued):

8. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.
9. If chlorine is present, add 2 drops of sodium thiosulfate solution, recap the bottle firmly and invert 5 times. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.
10. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C.
11. Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

V. SAMPLE TRANSPORT:

After obtaining the water samples in duplicate, attach the completed chain of custody seal around the plastic cap of each voa vial. Place each pair of vials into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use “blue ice” as the samples may not chill adequately. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. SAMPLE STORAGE:

All samples should be extracted as soon as possible after collection. Maximum holding time for extraction is 24 hours. Results of holding time studies suggest that all analytes with the possible exception of heptachlor were adequately stable for 14 days when transported and stored according to the above referenced conditions. In general, heptachlor showed inconsistent results. If heptachlor is to be determined, samples should be extracted within 7 days of collection.

VII. DEFINITIONS:

- A. *Sodium Thiosulfate* ($Na_2S_2O_3$): A preservative used to dechlorinate water samples. Reduces free chlorine into acid.
- B. *Blue ice*: Generic name for a non-toxic gel which when frozen is frequently used as a substitute for ice.

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 505: In the 40 ml vial which contains the sample, 5 ml of sample are removed from the vial and discarded. The sample is spiked with a surrogate and any other target compounds for quality control purposes. The spikes containing surrogate or target compounds are in either acetone or an alcohol matrix that forms an azeotroph with the water in the sample. The sample is “salted” out by adding 6 grams of sodium chloride and then 2 ml of hexane extraction solvent is added to the sample. Because water is a denser, polar substance and hexane is a lighter, nonpolar substance, the two immiscible liquids form a bilayer with hexane at the top. The sample is then recapped and mixed vigorously to complete the extraction process. The overall effect of this “salting” out procedure acts to “partition” or push the nonpolar organohalide pesticides and polychlorinated biphenyls (PCB's) analytes out of the water sample and into the nonpolar hexane extraction solvent. The hexane layer is removed from the sample and placed into a microvial.

Two μ l of the hexane extract is then injected into onto the column of a gas chromatograph for separation which is equipped with a linearized electron capture detector (ECD) for analysis and quantitation. The extraction time is 30 to 50 minutes per sample depending upon the analytical conditions selected. Confirmatory evidence can be obtained using a dissimilar column.